

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

CENTER FOR ENVIRONMENTAL MEASUREMENT AND MODELING RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF RESEARCH AND DEVELOPMENT

April 5, 2021

Ken Kloo, Director NJ Department of Environmental Protection Division of Remediation Management Mail Code 401-05M 401 East State Street P.O. Box 420 Trenton, NJ 08625-0420

Subject: NJDEP Report #9: Targeted and Non-targeted Analysis Results of PFAS in Soil Cores

Dear Mr. Kloo:

I am pleased to provide you with the attached laboratory report that includes targeted analysis (TA) and non-targeted analysis (NTA) results for per- and polyfluoroalkyl substance (PFAS) in soil core samples. This is the ninth in a series of reports prepared as a part of EPA Office of Research and Development's (ORD) collaboration with the New Jersey Department of Environmental Protection (NJ DEP) and EPA Region 2 on the study, "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey." The enclosed Report #9 provides results of the occurrence of legacy PFAS based on analysis with known standards and a group of chloroperfluoro-polyether-carboxylate congeners (ClPFPECAs) based on non-targeted analysis in 5 soil core samples.

It is our understanding that this information was requested by NJ DEP to help in their ongoing investigation into the presence of PFAS in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence that may be potentially associated with industrial releases. EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. We are providing the results of our analysis as they become available.

We do not interpret exposure or risk from concentrations of PFAS or ClPFPECAs in this report. The EPA does not currently have final health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided in the attached reports indicate the presence (or lack) of PFAS in the soil core samples, we do not have sufficient information to offer interpretations related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both EPA's and New Jersey's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrates EPA's commitment to cooperative federalism.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2107 or via email at Watkins.tim@epa.gov or Brian Schumacher at (706) 355-8001 or via email at Schumacher.Brian@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H. Watkins

Timothy H Watkins

Director

Enclosure

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Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey

Laboratory Data Report #9: Targeted and Non-targeted Analysis of PFAS in Soil Core Samples

Background. This report stems from a collaborative study with EPA ORD, Region 2, and NJ DEP entitled "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey". NJ DEP assumed responsibility for the collection of samples and their shipment to the ORD laboratory. ORD was responsible for sample extraction and analysis of PFAS. ORD's analysis and support team for this data report are listed in Table 1.

Table 1. EPA Office of Research and Development analysis and report team.

Responsibility	Personnel
ORD Principal Investigators	Andy Lindstrom, Mark Strynar, John Washington
Laboratory chemistry	Mary Davis, Marina Evich, John Washington, Tom Jenkins
Quality Assurance Review	Sania Tong-Argao
Management coordination and review	Brian Schumacher
Report preparation	Kate Sullivan, Mary Davis

This 9th report includes results of targeted and non-targeted analysis for PFAS in 5 soil core samples collected by NJ DEP on November 8 and 9, 2017, and delivered to the ORD laboratory in Athens, GA on November 14, 2017. The soil core samples were collected at 18 to 24" below ground surface at a subset of 5 of the 24 sites where surface soils were sampled. The results provided in this report were analyzed by Dr. Mary Davis under the direction of Dr. John Washington at ORD's laboratory in Athens, GA.

Thirteen perfluorinated carboxylic acids (PFCAs) and three perfluorinated sulfonates (PFSs) were quantitated using a targeted analysis approach. Characteristics of these compounds including the compound name, CAS registry number (CASRN), chemical formula, and monoisotopic mass are provided in Table 2. These same 16 PFAS are registered in EPA's CompTox Chemicals Dashboard¹ where additional information about these chemicals can be found.

Non-targeted analysis (NTA) was also performed on the soil core samples focusing on ten novel PFAS that are congeners of chloro-perfluoro-polyether-carboxylate (ClPFPECA). Table 3 provides the mass spectral features and molecular mass of the ClPFPECA congeners distinguished by carbon chain length and number of ethyl, propyl groups that were identified in the soil core samples with NTA.

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¹ U.S. EPA CompTox Chemicals Dashboard https://comptox.epa.gov/dashboard

 $\begin{tabular}{ll} Table 2. & PFCA and PFS Analyzed with Targeted Analysis in NJ Soil Core Samples by UPLC-MS \end{tabular}$

Acronym	Chemical Name	Formula	CAS Registry Number	Monoisotopic Mass (g/mol)	PFAS Type
PFBA	Perfluorobutanoic Acid	$C_4HF_7O_2$	375-22-4	213.9865	PFCA
PFPeA	Perfluoropentanoic Acid	$C_5HF_9O_2$	2706-90-3	263.9833	PFCA
PFHxA	Perfluorohexanoic Acid	C ₆ HF ₁₁ O ₂	307-24-4	313.9801	PFCA
PFHpA	Perfluoroheptanoic Acid	C ₇ HF ₁₃ O ₂	375-85-9	363.9769	PFCA
PFOA	Perfluorooctanoic Acid	C ₈ HF ₁₅ O ₂	335-67-1	413.9737	PFCA
PFNA	Perfluorononanoic Acid	C ₉ HF ₁₇ O ₂	375-95-1	463.9705	PFCA
PFDA	Perfluorodecanoic Acid	C ₁₀ HF ₁₉ O ₂	335-76-2	513.9673	PFCA
PFUnDA	Perfluoroundecanoic Acid	C ₁₁ HF ₂₁ O ₂	2058-94-8	563.9641	PFCA
PFDoDA	Perfluorododecanoic Acid	C ₁₂ HF ₂₃ O ₂	307-55-1	613.9609	PFCA
PFTrDA	Perfluorotridecanoic Acid	C ₁₃ HF ₂₅ O ₂	72629-94-8	663.9577	PFCA
PFTeDA	Perfluorotetradecanoic Acid	C ₁₄ HF ₂₇ O ₂	376-06-7	713.9545	PFCA
PFHxDA	Perfluorohexadecanoic Acid	C ₁₆ HF ₃₁ O ₂	67905-19-5	813.9482	PFCA
PFODA	Perfluorooctadecanoic Acid	C ₁₈ HF ₃₅ O ₂	16517-11-6	913.9418	PFCA
PFBS	Perfluorobutane Sulfonate	C ₄ HF ₉ SO ₃	375-73-5	299.9503	PFS
PFHxS	Perfluorohexane Sulfonate	C ₆ HF ₁₃ SO ₃	355-46-4	398.9366	PFS
PFOS	Perfluorooctane Sulfonate	C ₈ HF ₁₇ SO ₃	1763-23-1	499.9375	PFS

Table 3. Chloro-Perfluoro-Polyether-Carboxylate (CIPFPECA) Congeners Identified in Soil Cores Using Non-Targeted Analysis.

Carbon Chain Length	Anion Formula	Number of Ethyl, Propyl Groups	Molecular Mass of Anion (g/mol)		
7	C ₇ CIF ₁₂ O ₄	1,0	410.9294		
8	C ₈ CIF ₁₄ O ₄	0,1	460.9262		
9	C ₉ CIF ₁₆ O ₅	2,0	526.9179		
10	C ₁₀ CIF ₁₈ O ₅	1,1	576.9147		
11	C ₁₁ CIF ₂₀ O ₅	0,2	626.9115		
11	C ₁₁ CIF ₂₀ O ₆	3,0	642.9064		
12	C ₁₂ CIF ₂₂ O ₆	2,1	692.9032		
13	C ₁₃ CIF ₂₄ O ₆	1,2	742.9000		
13	C ₁₃ CIF ₂₄ O ₇	4,0	758.8949		
14	C ₁₄ CIF ₂₆ O ₆	0,3	792.8968		

METHODS IN BRIEF

The soil core samples were extracted and analyzed according to methods documented within an approved Quality Assurance Project Plan (QAPP)^{2,3}. These methods are also generally described in Washington et al. (2014, 2015, 2020).

In brief, each sample was divided into three ~l g aliquots and extracted individually. Samples were extracted with 90%:10% acetonitrile:water followed by a liquid/liquid cleanup. Extracts were first analyzed by ultra-performance liquid chromatography (UPLC)/mass spectrometry using a Waters Acquity UPLC coupled to a Waters Xevo quadrupole time-of-flight (QToF) mass spectrometer to identify the previously unknown PFAS. After these PFAS were identified, the extracts were quantitated via targeted analysis (TA) or semi-quantitated via NTA in separate batches.

Targeted Analysis

PFCA and PFS concentrations were determined using a targeted analysis approach based on authentic standard reference materials. Quantitation was based on mass-labeled internal multipoint calibration curves. These analyses were performed on sample aliquots, laboratory blanks, and check standards. Dilution of samples was not performed.

Non-targeted Analysis

Non-targeted analysis differs from targeted analysis in that chemical identification and quantification does not have the benefit of being based on an authentic standard for each compound.

PFAS concentrations were semi-quantitatively estimated by manual integration of chromatographic peaks in each of the 3 aliquot replicates followed by comparison of peak areas to a stable isotope-labeled compound, ¹³C₅-labeled perfluorononanoic acid (¹³C₅-PFNA), of known concentration that served as an internal standard (IS). CIPFPECA concentrations were estimated proportional to its peak area assuming the same instrument response as the labeled PFAS using Equation 1.

Equation 1.
$$ClPFPECA_{Conc} = \frac{clPFPECA_{PA} \times C^{13}PFNA_OA_{Conc}}{C^{13}PFNA_OA_{PA}}$$

where:

ClPFPECA_{Conc} is the semi-quantified ClPFPECA concentration (pg/g)

ClPFPECA_{PA} is the ClPFPECA peak area

 C^{13} -PFNA OA_{Conc} is the known concentration of labeled PFNA after spiking into the sample (pg/g)

² National Exposure Research Laboratory, Quality Assurance Project Plan: Detection, Evaluation and Assignment of Multiple Poly and Per-fluoroalkyl Substances (PFAS) in environmental media from an industrialized area of New Jersey. Prepared for New Jersey Department of Environmental Protection (NJDEP), D-EMMD-IEIB-010-QAPP-01, September 14, 2017.

³ National Exposure Research Laboratory, Quality Assurance Project Plan: Detection, Evaluation and Assignment of Multiple Poly and Per-fluoroalkyl Substances (PFAS) in environmental media from an industrialized area of New Jersey. Prepared for New Jersey Department of Environmental Protection (NJDEP), Amendment #1 D-EMMD-0031345-QP-1-1. May 2, 2018.

 C^{13} -PFNA OA_{PA} is the peak area of the labeled PFNA.

Our experience with PFAS suggests that this means of estimation is within an order of magnitude of the actual concentration. Even though the absolute concentration estimate will be uncertain, relative comparisons between samples for a given congener will be much less uncertain. Any application of NTA results should consider this inherently greater uncertainty with NTA than those performed by targeted laboratory analysis.

Limits of Detection and Quantitation

The limits of detection and quantification are determined using the same method for TA and NTA results. The limit of detection (LOD) and the limit of quantitation (LOQ) for sample concentrations are defined using a two-mean, one-tailed Student's t-test to verify a significant difference between chemical abundance averaged for the 3 aliquots and that observed in the laboratory process blanks³. This approach establishes unique limits for each sample arising from the sample-specific variation among the three aliquot replicates. Samples with no observed peak area in any aliquot are reported as Non-Detect ("ND"). A sample is reported as "<LOD" in the results tables if a peak area was observed in one or more of the 3 aliquots but the t-statistic for the aliquot replicates was less than $t_{critical at \alpha = 0.05}$. A sample is flagged as "<LOQ" if the t-statistic is greater than $t_{critical at \alpha = 0.05}$ but less than $t_{critical at \alpha = 0.01}$. We also do not report a value below the lowest standard of the calibration curves during targeted analysis, although usually the statistical calculation of the method detection limit based on the process blanks is the limiting qualification.

SUMMARY OF RESULTS

Results for analyte concentrations and quality assurance information are presented separately for the TA and NTA analyses.

Concentrations of PFCAs and PFSs Determined by Targeted Analysis

Targeted analysis results for the 5 soil core samples identified by sample IDs assigned by NJ DEP are provided in Table 4. The reported concentrations of PFCAs and PFSs are presented as the mean value of triplicate aliquot analysis and are adjusted to dry mass weight and corrected to the process-blanks (i.e., reported sample concentrations are analytical concentrations minus mean process blank values). The five soil core samples were collected with surface soil samples reported in NJ DEP Report #1⁴, with common locations sharing the same numeric portion of the sample ID (e.g., soil core sample PFSC008 was collected in the same location as surface soil sample PFSS008).

The highest concentrations of PFAS in the soil core samples were found for PFNA, PFUnDA, and PFOA. Sample PFSC001 and its duplicate PFSDUP2 had distinctly higher concentrations of these compounds than the other soil core samples. Generally, PFTeDA, PFHxDA, PFODA, PFBS, and PFHxS concentrations were at or below the LOQ with the exception of PFHxS in PFSCDUP2 and PFHxDA in sample PFSC017.

Data were checked for compliance with a number of laboratory and field related quality control evaluation criteria as specified in the project QAPP^{2,3}. Recovery was calculated for samples using the recovery internal standard, ¹³C₈-perfluorooctanoic acid (M8C₈), which was added to

⁴ NJDEP Laboratory Data Report #1: Targeted analysis of PFCA in Soil. U.S. EPA/ORD, January 31, 2019.

the field samples in known mass before extraction was initiated. The average recovery of M8C8 in the soil core samples was $90 \pm 4\%$. Recovery of the M8C8 was similar in the process blanks $(95 \pm 4\%)$.

The highest standard of the calibration range was 6,000 pg/g. Sample values exceeding this value are flagged as "JC1" in Table 4. The project QAPP specifies that back prediction of calibration curve points should be within \pm 30% for concentrations >15 pg/g and \pm 5 pg/g of the known concentration for concentrations <15 pg/g, with the least-squares calibration line maintaining central tendency³. Of the 80 check standard runs, 67 (84%) were within the acceptance criteria. Many of the check standard failures were observed in the concentration range that was less than the LOD and LOQ.

Nearly all of the solvent blanks were free of target compounds. Three solvent blanks presented concentrations that were apparently due to small peaks for internal standards. Samples were not remeasured as these small peaks did not carry through to the next samples in the analytical run and generally the blanks were clean. Two field blanks collected during the sampling trip were reported with the surface soil samples in NJ DEP Report #1⁴. These field blanks had measurable concentrations of PFCAs including PFPeA, PFHxA and PFHpA.

The coefficient of variation (CV) of the three aliquots analyzed for each analyte in each sample is a measure of the precision of laboratory measurements (equation 2).

Equation 2. Coefficient of Variation (%) = Standard Deviation /Mean x 100

The CV of 50 valid analyte/sample comparisons in which concentrations exceeded LOQ averaged 24% with all but 1 meeting the project acceptance goal of a CV<50%. Sample concentrations not meeting this criterion are flagged as "JP1" in Table 4.

The project QAPP² specifies that the relative percent different (RPD) of field duplicates should be <30%. Sample PFSC001 and its duplicate sample PFSCDUP2 met the criteria with an average RPD of analyte comparisons of 7.2% for the 8 valid comparisons.

Samples were processed for legacy perfluorinated compounds using targeted analysis 28 months after initial sample collection. This duration exceeded the holding time of 1 year specified in the project QAPP³.

Table 4. PFCA and PFS Concentrations (pg/g) in Soil Core Samples Determined with Targeted Analysis.

Carbon Length	C4	C5	C6	C7	C8	С9	C10	C11
Name	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA
Sample ID	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g
PFSC001	43.2 <loq< td=""><td>96.7 <loq< td=""><td>168</td><td>187</td><td>1,550</td><td>6,150 JC1</td><td>207</td><td>1,190</td></loq<></td></loq<>	96.7 <loq< td=""><td>168</td><td>187</td><td>1,550</td><td>6,150 JC1</td><td>207</td><td>1,190</td></loq<>	168	187	1,550	6,150 JC1	207	1,190
PFSCDUP2 ^{&}	<lod< td=""><td>130</td><td>168</td><td>180</td><td>1,970</td><td>6,780 JC1</td><td>206</td><td>1,010</td></lod<>	130	168	180	1,970	6,780 JC1	206	1,010
PFSC002	53.0 <loq< td=""><td>75.7</td><td>38.2 <loq< td=""><td>59.0</td><td>157 <loq< td=""><td>1,290</td><td>361</td><td>519</td></loq<></td></loq<></td></loq<>	75.7	38.2 <loq< td=""><td>59.0</td><td>157 <loq< td=""><td>1,290</td><td>361</td><td>519</td></loq<></td></loq<>	59.0	157 <loq< td=""><td>1,290</td><td>361</td><td>519</td></loq<>	1,290	361	519
PFSC008	35.2	43.6	38.9	48.8	60.4	1,640	413	501
PFSC017	87.1	269	249	297	813	353	186	76

Carbon Length	C12	C13	C14	C16	C18	S4	S6	S8
Name	PFDoDA	PFTrDA	PFTeDA	PFHxDA	PFODA	PFBS	PFHxS	PFOS
Sample ID	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g
PFSC001	<lod< td=""><td>143</td><td><lod< td=""><td>ND</td><td>ND</td><td><lod< td=""><td>4.20 <loq< td=""><td>118</td></loq<></td></lod<></td></lod<></td></lod<>	143	<lod< td=""><td>ND</td><td>ND</td><td><lod< td=""><td>4.20 <loq< td=""><td>118</td></loq<></td></lod<></td></lod<>	ND	ND	<lod< td=""><td>4.20 <loq< td=""><td>118</td></loq<></td></lod<>	4.20 <loq< td=""><td>118</td></loq<>	118
PFSCDUP2 ^{&}	19.6	141	<lod< td=""><td>ND</td><td>ND</td><td>11.0 <loq< td=""><td>8.00</td><td>121</td></loq<></td></lod<>	ND	ND	11.0 <loq< td=""><td>8.00</td><td>121</td></loq<>	8.00	121
PFSC002	20.1	55.0	9.35 <loq< td=""><td>ND</td><td>ND</td><td><lod< td=""><td><lod< td=""><td>212</td></lod<></td></lod<></td></loq<>	ND	ND	<lod< td=""><td><lod< td=""><td>212</td></lod<></td></lod<>	<lod< td=""><td>212</td></lod<>	212
PFSC008	11.5	58.0	<lod< td=""><td>ND</td><td>ND</td><td><lod< td=""><td>ND</td><td>286</td></lod<></td></lod<>	ND	ND	<lod< td=""><td>ND</td><td>286</td></lod<>	ND	286
PFSC017	55.9	37.8	34.6 <loq< td=""><td>14.1 JP1</td><td><lod< td=""><td><lod< td=""><td>ND</td><td>113</td></lod<></td></lod<></td></loq<>	14.1 JP1	<lod< td=""><td><lod< td=""><td>ND</td><td>113</td></lod<></td></lod<>	<lod< td=""><td>ND</td><td>113</td></lod<>	ND	113

QA/QC Flags

ND No peak area observed.

Peak area observed in one or more aliquots, but aliquot average not significantly greater than process blanks at T critical at $\alpha = 0.05$.

<LOQ</p>
Sample value less than the Limit of Quantitation determined for each sample: Tcalc of aliquots > Tcritical at α =0.05 but < Tcritical at α =0.01.

JP1 Sample aliquot triplicates do not meet acceptance criteria for precision.

 $_{
m JC1}$ Sample result exceeds the upper calibration range.

[&]Note that sample PFSCDUP2 is paired with sample PFSC001.

Semi-Quantitation of Chloro-Perfluoro-Polyether-Carboxylate (ClPFPECA) Concentrations Determined by NTA

Various members of the ClPFPECA congener series listed in Table 3 were previously found in soils and vegetation⁵, sediment⁶, and well water⁷,⁸ within the study area. The generic structure of the ClPFPECA congeners is shown in Figure 1. Analysis of surface soils⁵ identified nine congeners with ethyl and propyl groups (e,p) varying from 0 to 3.

Figure 1. Generic Structure of Chloro-Perfluoro-Polyether-Carboxylate (CIPFPECA), where e is the number of ethyl groups and p is the number of propyl groups

We have high confidence in the identification of the CIPFPECA congeners considering a combination of evidence including mass spectral data, consistency of our detection in other media (i.e., water, dispersions, soil, vegetation and sediment), and literature reports of Wang *et al.* (2013) establishing these PFAS in products produced by Solvay, a PFAS manufacturing company, that has a facility located within the geographic sampling area⁹.

Semi-quantitative concentration estimates for the CIPFPECA congeners expressed as ¹³C₅-PFNA are provided in Table 5. Sample values are reported as the mean of 3 aliquots that are corrected to the process-blanks, (i.e., reported sample concentrations are analytical concentrations minus mean process blank values), and are adjusted to dry weight.

⁵ NJDEP Report #2. Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey. Laboratory Data Report #2: Non-targeted Analysis of PFAS in Soil and Vegetation. U.S.EPA/ORD, March 8, 2019.

⁶ NJDEP Report #5. Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey. Laboratory Data Report #5: Non-targeted Analysis of PFAS in Sediment. U.S.EPA/ORD, April 23, 2020.

⁷NJDEP Report #7. Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey. Laboratory Data Report #7: Non-targeted Analysis of PFAS in in Water Samples Collected from Wells with GAC Treatment. U.S.EPA/ORD, April 23, 2020.

⁸ NJDEP Report #8. Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey. Laboratory Data Report #7: Non-targeted Analysis of PFAS in in Water Samples Collected from Water Samples Collected from Tidal and Non-tidal Surface Waters and Wells. U.S.EPA/ORD, September 23, 2020.

⁹ Washington, et al., 2020 Science DOI: 10.1126/science.aba7127

Soil core samples varied in abundance of CIPFPECA congeners, but one or more of the congeners was present in every sample in measurable concentrations. Most CIPFPECA congeners occurred at low concentrations except for C8 (0,1) and C10 (1,1) where they occurred at relatively higher concentrations in several of the samples (maximum concentration C8 (0,1) = 701 pg/g, C10 (1,1) =129 pg/g). As with previously reported concentrations in soils and vegetation (NJ DEP Report #2⁵) and sediment (NJ DEP Report #5⁶), the C8 (0,1) and C10 (1,1) congeners were the most abundant. On average, C8 (0,1) and C10 (1,1) constituted 84% and 11%, respectively, of the total signal of CIPFPECA congeners observed in the soil cores. CIPFPECA concentrations for the C8 (0,1) congener were very similar between the soil surface (NJ DEP Report #2) and soil core samples, but concentrations of other congeners were generally significantly lower than those reported in the surface soil samples.

Data were checked for compliance with a number of laboratory and field related quality control evaluation criteria as specified in the project QAPP^{2,3}, although QC measures are more limited for NTA. Precision of NTA results was checked by computing the Coefficient of Variation (CV) (eq. 2) among the 3 aliquots of each of the samples for each analyte. The CVs of the laboratory aliquots evaluate extraction and analytical precision and were within project goals of CV<50%³ in 100% of 24 valid analyte/sample comparisons (i.e., sample values>LOQ). Labeled internal standard concentrations can also be compared as a measure of laboratory reproducibility as the internal standard concentrations are the same in each check standard. The CV of the peak areas (reflective of concentration) for the six internal standards met the project goal of <50%, as all comparisons were less than 8.8% and had an average of 7.1%.

No ClPFPECA congeners were detected in the solvent blanks during NTA indicating no instrument contamination from these compounds. Two field blanks collected during soil sampling as previously reported in NJ DEP Report #2⁴ were also free of ClPFPECA congeners.

Results for the 1 duplicate pair (PFSC001 and PFSCDUP2) are provided in Table 5. The relative percent difference (RPD) of the field duplicate samples evaluates field and analytical precision. The duplicate pair was within project goals of RPD <30%² in 2 of 4 valid analyte/sample comparisons (i.e., sample values>LOQ) and one analyte (C7(1,0)) was not detected in either of the duplicates. The average RPD was 18.7%.

Samples were processed for novel perfluorinated compounds CIPFPECA 27 months after initial sample collection.

Table 5. Semi-Quantitative Concentrations of Chloro-Perfluoro-Polyether-Carboxylate (CIPFPECA) Congeners in Soil Core Samples Determined with Non-targeted Analysis Expressed as ¹³C₅-PFNA, in pg/g.

Carbon Length	7	8	9	10	11	11	12	13	13	14
Ethyl, Propyl Groups	1,0	0,1	2,0	1,1	0,2	3,0	2,1	1,2	4,0	0,3
Soil Coil Sample ID		Concentration of CIPFPECA as M5PFNA (by simple ratios to matrix internal standard in pg/g soil)								
PFSC001	ND	701	4.85	129	28.9	1.71	4.55	ND	1.21	1.70
PFSCDUP2 ^{&}	ND	665	4.80	93.2	20.1	<lod< td=""><td>1.71 <loq< td=""><td><lod< td=""><td><lod< td=""><td><lod< td=""></lod<></td></lod<></td></lod<></td></loq<></td></lod<>	1.71 <loq< td=""><td><lod< td=""><td><lod< td=""><td><lod< td=""></lod<></td></lod<></td></lod<></td></loq<>	<lod< td=""><td><lod< td=""><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td><lod< td=""></lod<></td></lod<>	<lod< td=""></lod<>
PFSC002	ND	86.6	1.06	11.1	2.53 <loq< td=""><td>0.33 <loq< td=""><td>0.53 <loq< td=""><td><lod< td=""><td>0.64 <loq< td=""><td>ND</td></loq<></td></lod<></td></loq<></td></loq<></td></loq<>	0.33 <loq< td=""><td>0.53 <loq< td=""><td><lod< td=""><td>0.64 <loq< td=""><td>ND</td></loq<></td></lod<></td></loq<></td></loq<>	0.53 <loq< td=""><td><lod< td=""><td>0.64 <loq< td=""><td>ND</td></loq<></td></lod<></td></loq<>	<lod< td=""><td>0.64 <loq< td=""><td>ND</td></loq<></td></lod<>	0.64 <loq< td=""><td>ND</td></loq<>	ND
PFSC008	ND	263	1.74	28.2	8.71	0.68	1.53	<lod< td=""><td>0.82</td><td><lod< td=""></lod<></td></lod<>	0.82	<lod< td=""></lod<>
PFSC017	<lod< td=""><td>6.62</td><td><lod< td=""><td>0.86</td><td>ND</td><td>ND</td><td>ND</td><td>ND</td><td>0.59 <loq< td=""><td>ND</td></loq<></td></lod<></td></lod<>	6.62	<lod< td=""><td>0.86</td><td>ND</td><td>ND</td><td>ND</td><td>ND</td><td>0.59 <loq< td=""><td>ND</td></loq<></td></lod<>	0.86	ND	ND	ND	ND	0.59 <loq< td=""><td>ND</td></loq<>	ND

QA/QC Flags

ND No peak area observed

 \leq LOD Peak area observed in one or more aliquots, but aliquot average not significantly greater than process blanks at T critical α = 0.05

 $^{<}$ LOQ Sample value less than Limit of Quantitation determined for each sample: Tcalc of aliquots > Tcritical at α=0.05 but < Tcritical at α=0.01

[&]Note that sample PFSCDUP2 is paired with sample PFSC001

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